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During the one year period of this grant October 15, 1987-October 14, 1988, efforts on the Program "Superconductivity and Magnetism in Layered Materials" were largely focused on introduction of our new program in high temperature superconductivity. We also completed studies of magnetic and superconducting graphite intercalation compounds.

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AFOSR Grant #88-0021
for the period
October 15, 1987 — October 14, 1988

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Contents

1 Abstract

During the one year period of this grant October 15, 1987—October 14, 1988, efforts on the program "Superconductivity and Magnetism in Layered Materials" were largely focused on introduction of our new program in high temperature superconductivity. We also completed studies of magnetic and superconducting graphite intercalation compounds. With regard to our research in high T_c superconductivity, summary reports are given for our projects on the infrared anisotropy of $La_{1.85}Sr_{0.15}CuO_{4-y}$, optical studies and characterization of superconducting thin films of bismuth strontium calcium copper oxide, observation of a spin density wave in La_2CuO_4 by Raman scattering, the effect of microstructure on the vibrational, electrical, and structural properties of $La_{1.85}Sr_{0.15}CuO_{4-y}$, and the surface impedance of superconducting $YBa_2Cu_3O_7$ in a magnetic field. Also reported are summaries of research in graphite intercalation compounds including a study of magnetic phase transitions in acceptor compounds from anomalies in the transport properties, high field magnetization of stage 1 $CoCl_2$–GICs, high resolution microscopy studies of $MnCl_2$–GICs, anisotropic superconductivity in C_4KHg, and surface phenomena in donor and acceptor graphite intercalation compounds.

2 Summary of Research Effort

A summary of the status of the research effort on "Superconductivity and Magnetism in Layered Materials" is presented in terms of the progress made during the period October 15, 1987 to October 14, 1988. During this time period we were phasing out a previous AFOSR program on superconductivity and magnetism in graphite intercalation compounds and phasing in a research program on superconductivity and magnetism in high T_c superconducting materials. Consequently, seed funding for some of the high T_c collaborative work with the Lincoln-RADC-MIT consortium members came from other funding sources. By the end of the funding period the conversion from the old program to the new program was completed. Publications pertinent to this research are listed in §3.1 and §3.2.

2.1 Research in High T_c Superconductivity

2.1.1 Infrared Anisotropy of $La_{1.85}Sr_{0.15}CuO_{4-y}$

Analysis of the optical properties of the high T_c superconductors is complicated by the high degree of anisotropy of these materials. Because of the difficulty in preparing single crystals of the high T_c materials with large enough volume to carry out a-face and c-face optical studies, many of our optical studies have been on polycrystalline samples prepared by members of the consortium. By calculating the infrared reflectance $R(\omega)$ for a collection of randomly oriented, highly anisotropic crystallites, we have been able to fit the measured reflectance of polycrystalline $La_{1.85}Sr_{0.15}CuO_{4-y}$ samples which are superconducting at low temperatures. From this measurement and analysis the room-temperature tetragonal $La_{1.85}Sr_{0.15}CuO_{4-y}$ samples are found to be metallic in the Cu-O planes and nonmetallic out-of-plane. The deconvolution of $R(\omega)$ into $R_{\|}$ and $R_{\perp}$ allows the anisotropy of the system to be examined and provides a method by which infrared measurements of polycrystalline materials can be interpreted. These results on a range of $La_{2-x}Sr_xCuO_{4-y}$ samples were later confirmed by polarized reflectivity measurements carried out on single crystals of the $x = 0$ material La_2CuO_{4-y} which did not show a superconducting phase.

2.1.2 Optical Studies and Characterization of Superconducting Thin Films of Bismuth Strontium Calcium Copper Oxide

We have studied the lattice vibrations and electronic structure of superconducting thin films of $Bi_2Sr_2Ca_1Cu_2O_{8+x}$ prepared by members of the consortium with Raman scattering and optical absorption spectroscopies. Films having thicknesses of 0.5 - 1.5 μm and $T_c = 74$K were deposited by consortium members using a reactive magnetically-enhanced triode sputtering technique onto substrates of Al_2O_3, $SrTiO_3$, and yttria-stabilized zirconia. The textured films were found to be oriented with the c-axis perpendicular to the substrate which enables the optical properties to be studied independent of the crystalline anisotropy. We have obtained the frequency dependent dielectric functions over the wavelength range 3200 - 300 nm from a Kramers-Kronig analysis of the room-temperature $\vec{E}\perp\hat{c}$ absorption spectra, and have examined the Raman-allowed A_g phonons and high frequency excitations in the Raman spectra. A comparison has been made of the optical properties of films deposited on the different substrates, as well as with the optical properties of the $YBa_2Cu_3O_{7-\delta}$ and $La_{2-x}Sr_xCuO_{4-y}$ materials studied by us and by other workers.

2.1.3 Observation of a spin density wave in La_2CuO_4 by Raman scattering

In certain samples of La_2CuO_4 a multi-peaked Raman spectrum has been observed in the region from $\sim$300 cm^{-1} to $\sim$1420 cm^{-1}. We have observed a striking example of such a spectrum from room temperature to $\sim$500 K in a single-crystal sample prepared at the MIT Center for Materials Science and Engineering. In collaboration with Dr. H.J. Zeiger of Lincoln Laboratory, this unusual spectrum has been explained on the basis of the allowed excitation of spin wave modes in the presence of a spin density wave. In particular, a good fit to the observed spectral frequencies is obtained in terms of a long-wavelength spiral spin configuration. The role of the dynamic spin correlation length in broadening the spin-wave spectrum and in affecting the temperature and sample dependence of the spectrum has been considered. Analysis of the Raman spectrum leads to an unusual spin-wave spectrum with important implications for low temperature specific heats and superconductivity in the cuprate superconductors. This work is continuing both experimentally and theoretically.

2.1.4 Effect of microstructure on the vibrational, electrical, and structural properties of $La_{1.85}Sr_{0.15}CuO_{4-y}$

Transmission electron microscopy and scanning Auger spectroscopy studies of polycrystalline $La_{1.85}Sr_{0.15}CuO_{4-y}$ have been carried out to characterize polycrystalline samples prepared by consortium members and used for our optical and Raman studies. The microscopic measurements provide an opportunity for a detailed examination of the intra- and inter-granular compositional differences as a function of oxygen concentration. Superconducting transitions of the polycrystalline samples which are observed to vary with grain sizes and oxygen concentration, have been correlated with the microscopy results providing estimates of the Josephson tunneling between grains.

2.1.5 Surface Impedance of Superconducting $YBa_2Cu_3O_7$ in a Magnetic Field

We report the results of measurements of the surface impedance $Z = R_s + iX_s$ of $YBa_2Cu_3O_7$ using a 16.5 GHz gold plated cylindrical cavity operating in a TE_{011} mode. Two sintered ceramic $YBa_2Cu_3O_7$ samples ($T_c \simeq 90$ K) prepared by members of the consortium were used as cavity end plates and a magnetic field H (up to 9 T) was applied parallel to the sample surface. The surface resistance R_s increased rapidly as the magnetic field increased from 0 to 0.7 T. For higher fields, we observed a slower monotonic rise in $R_s(H)$. The measured surface resistance near T_c was used to determine the functional form of the upper critical field $H_{c2}(T)$. The temperature dependence of the surface resistance at zero field was also used to characterize the samples.

2.2 Research in Graphite Intercalation Compounds

2.2.1 Study of Magnetic Phase Transitions in Acceptor Compounds from Anomalies in the Transport Properties

This work on magneto-transport in magnetic GICs was completed as Nai-Chang Yeh completed her Ph.D. thesis. Our previous studies had shown that the strong coupling between graphite layers and Eu layers in the donor compound C_6Eu results in strong spin scattering effects on the graphite conduction π-electrons by the magnetic excitations of the Eu ions. Therefore dramatic changes in the magnetoresistance of C_6Eu are observed as the system undergoes magnetic phase transitions. Because of the strong interaction, the magnetic phase transitions occur at very high magnetic fields (from several tesla to over 20 tesla). In contrast, the acceptor-type magnetic GICs (which were studied during the past year) have a much weaker coupling between the intercalate and the graphite layers because of the much larger spatial separation between the graphite conduction electrons and the magnetic species. Therefore the change in the magnetoresistance due to changes in magnetic ordering is expected to be small and to occur at much lower fields. This is indeed found to be the case.

$CoCl_2$-GICs are well known as 2D-XY systems with a strong ferromagnetic in-plane nearest neighbor coupling and a relatively weak antiferromagnetic interplane coupling. From magnetic susceptibility and Monte Carlo studies that we have previously carried out, stage-1 $CoCl_2$-GIC samples are known to exhibit three magnetic phases at low temperatures. For zero magnetic field, adjacent planes of ferromagnetically ordered Co^{2+} spins (superspin planes) are antiferromagnetically ordered with respect to each other. At a lower critical field, these superspin planes undergo a spin flop transition and the superspins align along easy axes, making angles of 120° with respect to each other. At a somewhat higher field a transition is made to a spin aligned paramagnetic state, whereby all spins are aligned along the externally applied magnetic field. In zero magnetic field two distinct peaks in the magnetic susceptibility of the stage 1 $CoCl_2$-GIC are observed, at $T_{c1} \sim 8.3$ K and $T_{c2} \sim 9.7$ K, corresponding to the onset of magnetic order.

The zero-field temperature-dependent resistivity measurements (completed during the past year) for stage-1 and stage-2 $CoCl_2$-GICs each show anomalous behavior. For the stage 1 compound, the anomalous behavior at the magnetic phase transition is large, with the change in resistivity at the Néel temperature showing an opposite sign to what is typically observed in magnetic spin systems. At high temperatures, $T \gg T_c$, the temperature dependence of the resistivity obeys the standard functional form $\rho(T) = A + BT + CT^2$ typical of GICs. Below about 25 K, $\rho(T)$ shows less T dependence than would be expected from the standard functional form, and as T decreases below T_{c2}, a sharp <u>increase</u> in $\rho(T)$ is observed. Spin disorder scattering, characteristic of magnetic systems in the paramagnetic phase, typically result in a lowering of $\rho(T)$ below the magnetic ordering temperature. This effect has been explained in terms of Fermi surface changes associated with a doubling of the magnetic unit cell as antiferromagnetic alignment of the superspin planes is established. Theoretical models for these effects have been developed and they account well for the experimental magnetoresistance measurements.

Interesting behavior is also observed in the stage 1 compound as a function of magnetic field. Below T_{c1}, the resistivity $\rho(H)$ is almost independent of H until a critical field is reached corresponding to the alignment of the superspin planes along the magnetic field. Above this critical field, the resistivity $\rho(H)$ is observed to decrease and to saturate at much higher fields. The temperature dependence of these phenomena has been studied experimentally and a model has been developed to explain the observed field dependence in the various magnetic phases.

The behavior of $\rho(T, H)$ for the stage 2 $CoCl_2$–GIC is qualitatively different from that in stage 1 with regard to both sign and magnitude. As a function of temperature, the stage 2 compound shows a decrease in $\rho(T, 0)$ as T falls below T_{cl}, as expected from the enhanced spin disorder scattering above T_{cl}. The magnitude of the discontinuities in $\rho(T, H)$ at the magnetic phase transitions is much smaller for the stage 2 compound relative to stage 1, as expected from the much weaker interplanar exchange coupling. Model calculations have been made to account for the relative magnitudes observed for the stage dependence of the magnetoresistance.

Published papers by N.-C. Yeh are cited in §3.2.

2.2.2 High Field Magnetization of Stage 1 $CoCl_2$–GICs

The high field magnetization of stage 1 $CoCl_2$–GICs was studied as a function of field up to 18 tesla. Of particular interest has been the hysteretic behavior that has been observed well above the magnetic phase transition, indicating that there is no remanent magnetization. High field magnetization measurements provide important information on the magnetic parameters for the intercalate layer.

2.2.3 High Resolution Microscopy Studies of $MnCl_2$–GICs

An in-depth high resolution transmission electron microscopy (TEM) study of magnetic GICs has been carried out by Jim Speck. Particular emphasis has been given to determining the relation of the incommensurate intercalate structure to that of the adjacent graphite layers. A second focus of the work has been directed to the structure and temperature dependence of the intercalate domains. Four valuable pieces of information have been obtained from the TEM diffraction patterns: (1) The in-plane lattice parameters in the metal chloride sandwich are within 1% of the values in the pristine material. (2) The intercalate sandwich is translationally incommensurate with respect to the carbon lattice, but it is orientationally locked to the carbon. (3) Qualitative kinematical intensity calculations show that the chloride layer has $\bar{3}$ symmetry which is identical to the close packed layer in the pristine $MnCl_2$. (4) Careful examination of the intercalate reflections reveals a clustering of reflections within a small angular spread, but all for the same intercalate lattice constants. This slight orientational rotation between chloride layers leads to Moiré fringe images in dark field TEM images.

Jim Speck is nearing completion of his Ph.D. thesis. Support for his effort was switched to another grant in mid-year. Publications pertinent to this work are listed in §3.2.

2.2.4 Anisotropic Superconductivity in C_4KHg

Two superconducting transition temperatures, 0.8 K and 1.5 K, are reported for the stage 1 potassium-mercury graphite intercalation compound, C_4KHg. Structurally, the β phase is consistently found in the lower-T_c samples, while the higher-T_c samples always contain only the α phase. Adding a minute amount of hydrogen to the $T_c = 0.8$ K samples raises their transition temperature to 1.5 K. Measurements of the angular dependence of the critical field suggest that the higher-T_c samples have type I character for applied field orientations close to the c–axis. The temperature dependence of the upper critical field of C_4KHg shows extended linearity for both types of samples. The critical field data of C_4KHg and other GICs has been discussed in light of multiband and anisotropic Fermi surface models of superconductivity. The thermodynamic parameters obtained from the critical field data have been compared to the specific heat data measured by other workers. The hydrogen-induced enhancement of T_c is tentatively explained by a charge-density wave hypothesis. Considerable attention has been given to relating the anisotropy in H_{c2} for the graphite intercalation compounds to that found in the high T_c materials. Alison Chaiken completed her Ph.D. thesis in December of 1988 on a "Study of the Superconducting Properties of Ternary Graphite Intercalation Compounds".

2.2.5 Surface Phenomena

A visit by Dr. Michel Laguës from Paris stimulated investigation of whether or not there is intercalate depletion in the surface layer of acceptor GICs and intercalate enhancement for donor GICs. In collaboration with Laguës, XPS and Auger measurements have been carried out at MIT in the acceptor $CoCl_2$–GICs and $CuCl_2$–GICs. The results support the intercalate depletion for these acceptor compounds. Previous work on the donor Cs–GICs indicated intercalate enhancement. A model to explain the near surface behavior in donor and acceptor GICs was developed as a consequence of these experimental studies.

3 Reports and Publications

3.1 Publications on Phasing out of Graphite Intercalation Compound Research

1. M. S. Dresselhaus, N. C. Yeh, K. Sugihara, J. T. Nicholls, and G. Dresselhaus. Transport studies of magnetic graphite intercalation compounds. In D. Guérard and P. Lagrange, editors, *International Colloquium on Layered Compounds*, page 199, (1988). Pont-a-Mousson Conference.

2. A. Chaiken, T. P. Orlando, P. M. Tedrow, and G. Dresselhaus. Critical field measurements on superconducting graphite–KHg multilayers. In T. W. Barbee, Jr., F. Spaepen, and L. Greer, editors, *Multilayers: Synthesis, Properties, and Nonelectronic Applications*, page 295, Materials Research Society, Pittsburgh, PA, (1988). volume **103**.

3. N. C. Yeh, K. Sugihara, M. S. Dresselhaus, and G. Dresselhaus. Transport properties and magnetic exchange mechanisms of acceptor–type magnetic graphite intercalation compounds. *Phys. Rev.*, **B40**, (1989). (in press).

4. K. Sugihara, N. C. Yeh, M. S. Dresselhaus, and G. Dresselhaus. Transport properties in $CoCl_2$–graphite intercalation compounds. *Phys. Rev.*, **B39**, 4577, (1989).

5. M. S. Dresselhaus. New trends in intercalation compounds. *Mater. Sci. Eng.*, 1, 259, (1988). Section B: Solid State Materials for Advanced Technology.

6. N. C. Yeh, K. Sugihara, M. S. Dresselhaus, and G. Dresselhaus. Magnetic susceptibility studies of graphite intercalation compounds. *Phys. Rev.*, **B38**, 12615, (1988).

7. M. S. Dresselhaus. Intercalation compounds and composites. *Il Nuovo Cimento*, xxx, (1988). Enrico Fermi School.

8. J. S. Speck, B. J. Wuensch, and M. S. Dresselhaus. Structure of stage 1 and 2 metal–dichloride–GICs. In M. Endo, M. S. Dresselhaus, and G. Dresselhaus, editors, *Extended Abstracts of the Symposium on Graphite Intercalation Compounds at the Materials Research Society Meeting, Boston*, page 29, Materials Research Society Press, Pittsburgh, PA, (1988).

9. R. Al-Jishi, A. Chaiken, and M. S. Dresselhaus. Superconductivity in graphite intercalation compounds. In M. Endo, M. S. Dresselhaus, and G. Dresselhaus, editors, *Extended Abstracts of the Symposium on Graphite Intercalation Compounds at the Materials Research Society Meeting, Boston*, page 53, Materials Research Society Press, Pittsburgh, PA, (1988).

10. J. T. Nicholls, J. S. Speck, and G. Dresselhaus. Magnetic and structural properties of stage-1 $NiCl_2$-GICs. In M. Endo, M. S. Dresselhaus, and G. Dresselhaus, editors, *Extended Abstracts of the Symposium on Graphite Intercalation Compounds at the Materials Research Society Meeting, Boston*, page 77, Materials Research Society Press, Pittsburgh, PA, (1988).

11. J. T. Nicholls, J. S. Speck, and G. Dresselhaus. Magnetic and structural properties of stage-1 $NiCl_2$-GICs. *Phys. Rev.*, **B39**, xxx, (1989). (in press).

12. A. Chaiken and M. S. Dresselhaus. Temperature dependence of the critical field in superconducting GICs. In *Extended Abstracts of the 19th Biennial Conference on Carbon*, page xxx, (1989). July 19-24, 1989, State College, PA.

13. N.-C. Yeh, K. Sugihara, M.S. Dresselhaus and G. Dresselhaus, "**Magnetic Scattering Effects and Magnetic Exchange Mechanisms of Transition-Metal-Chloride GICs**", *Bull. American Physical Society* **33**, 729 (1988).

14. J.T. Nicholls, G. Dresselhaus, Y. Iye and K. Miura, "**Magnetostriction of Stage-1 $CoCl_2$-GICs**", *Bull. American Physical Society* **33**, 729 (1988).

15. A. Kazeroonian, M.S. Dresselhaus, A. R. Kortan, "**Phase transformations in stage-2 KHg-GICs**", *Bull. American Physical Society* **33**, 428 (1988).

3.2 Publications on MIT–RADC–Lincoln Lab Consortium on High T_c Superconductivity

1. G. L. Doll, J. T. Nicholls, M. S. Dresselhaus, A. M. Rao, J. M. Zhang, G. W. Lehman, P. C. Eklund, G. Dresselhaus, and A. J. Strauss, "**Effects of Sr doping on the ir-active phonons, plasmons, and interband transitions in polycrystalline $La_{2-x}Sr_xCuO_{4-y}$**", *Phys. Rev.*, **B38**, 8850, (1988).

2. J.S. Speck, G.L. Doll, M.S. Dresselhaus, G. Dresselhaus, A.J. Strauss and H.J. Zeiger, "**Effect of Microstructure on the Vibrational, electrical and Structural Properties of $La_{1.85}Sr_{0.15}CuO_{4-y}$**", *Bull. American Physical Society* **33**, 648 (1988).

3. G.L. Doll, I. Ohana, M.S. Dresselhaus and G. Dresselhaus, "**Optical Phonon Frequencies and Mode Assignments of $La_{2-x}Sr_xCuO_4$**", *Bull. American Physical Society* **33**, 417 (1988).

4. I. Ohana, Y.C. Liu, M.S. Dresselhaus, G. Dresselhaus, P.J. Picone, H.P. Jenssen, D.R. Gabbe, A.J. Strauss and H.J. Zeiger, "**Raman Scattering from Spin States and Phonons in Single Crystals of $La_{2-x}Sr_xCuO_4$**", *Bull. American Physical Society* **33**, 417 (1988).

3.3 Advanced Degrees and Honors

- M.S. Dresselhaus
  Annual Achievement Award, Engineering Societies of New England, 1988

- M.S. Dresselhaus
  Doctorat Honoris Causa, Université Catholique de Louvain, Louvain-la-Neuve, Belgium, 1988

- Y.C. Yeh, Ph.D., Department of Physics, January 1988.
  "Electronic and Magnetic Properties of Graphite Intercalation Compounds".

- A. Chaiken, Ph.D., Department of Physics, December 1988.
  "The Superconducting Properties of Ternary Graphite Intercalation Compounds".

4 Personnel Involved with Research Program

- Mildred Dresselhaus – Principal Investigator.
  Responsible for the research and the direction of all aspects of the program.

- Gene Dresselhaus – Co-principal Investigator.
  Responsible together with the principal investigator for the research and the direction of all aspects of the program.

- G. Doll – Postdoctoral Associate.
  Responsible for infrared and optical measurements of high T_c superconductors.

- I. Ohana – Postdoctoral Associate.
  Responsible for the study of the Raman spectra in high T_c superconductors.

- Alison Chaiken – Research Assistant.
  Responsible for superconductivity studies in intercalated graphite, and synthesis, characterization and properties measurements on these compounds. Also studies relation of anisotropic GIC superconductors to high T_c superconductors. Completed Ph.D. in December 1988 (now at NRL).

- Chi-Chung Chin – Research Assistant.
  Responsible for microwave studies in high T_c superconductors.

- A. Kazeroonian – Research Assistant.
  Responsible for studies of structural phase transitions in graphite intercalation compounds exhibiting superconductivity. Also working on optical studies of high T_c superconductors.

- J. Nicholls – Research Assistant
  Responsible for low dimensional susceptibility and magnetization studies of magnetic graphite intercalation compounds and related Monte Carlo model calculations. (Phased out of program.)

- James Speck – Research Assistant.
  Responsible for structural studies of intercalated graphite using x–ray diffraction, electron diffraction, real space imaging and lattice fringing, with particular emphasis on the orientational locking phenomenon. (Phased out of program.)

- N.C. Yeh – Research Assistant.
  Responsible for transport studies of low dimensional magnetism in magnetic graphite intercalation compounds. Completed Ph.D. in January, 1988 (now at IBM).

5 New Discoveries, Patents or Inventions

None.